Originator: **Kenyon C. Carlson, Manager**
ADEQ QA Unit

Contact For Information: **Kenyon C. Carlson, Manager**
ADEQ QA Unit

METHOD 550

I. SCOPE AND APPLICATION:

This method describes the procedure for determining specific polycyclic aromatic hydrocarbons (PAHs) in drinking water sources and finished drinking water. The following compounds can be determined using this method.

Analyte	Chemical Abstract Services Registry Numbers (CASRN)
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo(a)anthracene	56-55-3
Benzo(a)pyrene	50-32-8
Benzo(b)fluoranthene	205-99-2
Benzo(g,h,i)perylene	191-24-2
Benzo(k)fluoranthene	207-08-9
Chrysene	218-01-9
Dibenzo(a,h)anthracene	53-70-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Indeno(1,2,3-cd)pyrene	193-39-05
Naphthalene	91-20-3
Phenanthrene	85-01-8
Pyrene	129-00-0

II. REAGENTS:

- Sodium Thiosulfate ($Na_2S_2O_3$) solution
- 1:1 hydrochloric acid solution

III. MATERIALS:

- 1-liter amber borosilicate sample bottle fitted with screw caps lined with Teflon
- Latex gloves
- Paper towels
- Plastic container for disposal of used pipette tips
- Disposable glass pipette and rubber bulb.
- Pliers and protective eyewear

IV. PROCEDURE:

1. Remove any attachments such as hoses, screens or aeration devices on the faucet. Inspect the faucet for anything that may fall into the sample container.

2. Open the tap and allow the system to flush for about 10 minutes. This should be sufficiently long enough to allow the water temperature to stabilize and get a representative sample.

3. Adjust the water flow to about 1000 ml/minute or slow enough that no air bubbles purge the sample when collecting from the flowing stream.

4. Remove the cap from the 1-liter container. Do not rinse the container as it has already been acid rinsed and may already contain sodium thiosulfate as a preservative.

5. To fill, tip the bottle to about a 45° angle into the stream of water. Ensure the stream is sufficiently slow so as to be able to anticipate when the bottle is nearly full and thus avoid over flowing. Fill the bottle to within approximately 1 inch of its mouth.

.

6. Remove the bottle from the flow and recap. Invert the sample bottle five times.

7. Place a chlorine detector strip on a dry opened paper towel. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Thoroughly moisten the chlorine detector strip with the aliquot from the glass pipette and immediately flick the chlorine detector strip once using a sharp wrist motion to shake off the excess water. A determination must be made within 30 seconds.

8. If no chlorine is detected, recap the bottle firmly, dry the sample bottle, attach the sample/laboratory label to the bottle and secure the chain of custody seal around the cap. Record the results in the field notebook and place the sample bottle in the ice chest to cool to 4°C.

IV. PROCEDURE (continued):

9. If chlorine is present, add 5 drops of sodium thiosulfate solution, recap the bottle firmly and invert 5 times. Place a chlorine detector strip on a dry opened paper towel. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Moisten the chlorine detector strip with the aliquot from the glass pipette and immediately flick the chlorine detector strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference chlorine range. A determination must be made within 30 seconds.

10. If no chlorine is detected, recap the bottle firmly, dry the sample bottle, attach the sample label to the bottle and secure the chain of custody seal around the cap. Record the results in the field notebook and place the sample bottle in the ice chest to cool to 4°C.

11 Continue the process of adding sodium thiosulfate to the sample, recapping, mixing, and testing until no chlorine is detected. Remember to note the number of drops of sodium thiosulfate added to the water sample in the field notebook.

12. To preserve the sample by reducing the pH to $\leq$ 2 in order to inhibit microbiological activity, add 3 mls of 1:1 hydrochloric acid (HCl), recap the bottle firmly and invert 5 times. Tear off another inch strip of pH indicator paper and place on a dry portion of the paper towel.

13. Uncap the sample bottle and remove an aliquot of the sample using a clean glass pipette. Thoroughly moisten the pH indicator strip using the aliquot obtained in the glass pipette and immediately flick the pH strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference pH range within 30 seconds.

14. If the pH is $\leq$ 2, recap the bottle firmly, dry the sample bottle, attach the sample/laboratory label to the bottle and secure the chain of custody seal around the cap. Record the results in field notebook and place the sample bottle in the ice chest to cool to 4°C.

15. Continue the process of adding acid to the sample, recapping, mixing, and testing until the pH of the sample reaches a pH of $\leq$ 2. Remember to note the amount of acid added to the water sample in the field notebook.

V. SAMPLE TRANSPORT:

After obtaining the water samples, attach the completed chain of custody seal around the plastic cap of each 1-liter bottle. The 1-liter bottle must be amber colored to reflect sunlight since PAH's are known to be light sensitive, therefore samples should be stored in amber bottles or foil-wrapped in order to minimize photolytic decomposition. Place the sample bottle into the ice chest for transport. The samples must be chilled and preserved at a temperature of 4°C and maintained at that temperature from the time of collection until analysis. All samples must be extracted within 7 days of collection and completely analyzed within 30 days of extraction. Always use chopped, grated, or dry ice when chilling the voa samples for transportation. Never use "blue ice" as the semi-volatile samples may not chill adequately. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure they will be at 4°C upon arrival at the laboratory.

VI. SAMPLE PRESERVATION:

A. *Sodium thiosulfate ($Na_2S_2O_3$) solution*

B. *1:1 hydrochloric acid:* Add 500 ml of concentrated ultra pure hydrochloric acid to 400 ml of ASTM type I water (reagent water-- free of analytes) and dilute to a volume of 1 liter.

VII. DEFINITIONS:

A. *Sodium Thiosulfate ($Na_2S_2O_3$):* A preservative use to dechlorinate water samples that reduces free chlorine into acid.

B. *Aliquot:* A subsample or portion of a substance.

VIII. SAFETY:

The use of protective eyewear and laboratory quality latex gloves is highly recommended when collecting and preserving samples.

IX. SUMMARY OF METHOD:

METHOD 550: Approximately 1-liter of sample is serially extracted with methylene chloride using a separatory funnel. The methylene chloride extract is evaporated and concentrated to a volume of 1 ml. A 3.0 ml portion of acetonitrile is added to the extract and concentrated to a final volume of 0.5 ml. The extract analytes are then separated using high performance liquid chromatography (HPLC). Ultraviolet adsorption (UV) and fluorescence detectors are used in conjunction with HPLC to quantitatively measure the PAHs.